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REPORT ON LABORATORY EXPERIMENTS ON PERLON PRODUCTION METHODS IN THE GDR

50X1-HUM

Perfection of Process for Continuous Polymerization and Spinning of Perlon (Vacuum Process)

Continuous evacuation of liquid perlon (perlon melt) containing lactam is rejected in trade circles as operationally not feasible. In spite of this, we did work on this subject in order to test out again all possibilities of continuous evacuation and to initiate appropriate experiments.

In these experiments it developed that in addition to the discoloration of the liquid material resulting from air which was sucked in, considerable difficulties were encountered in that the bubbly liquid under vacuum could not be picked up by the pumps. Even a substantially satisfactory arrangement of the pressure pump (see quarterly report II/1951) was not adequate to pick up enough of the material being held back by the vacuum to make possible a satisfactory spinning process. The number and placement of the agitator arms and scrapers attached to the pump's drive shaft to force the liquid material into the pressure pump were variously altered without achieving the desired result. An attempt was also made to force the material under vacuum into the pump by means of a spiral arrangement of the agitator arms, but experiments in this connection also failed to produce the desired result.

However, these experiments ought to be continued in a different form. At present, a trial arrangement is being designed in which the liquid material under vacuum is to be forced into the geared pump by means of a plunger. We feel that another possibility for continuous evacuation and spinning of liquid material under vacuum is the use of only a moderate vacuum and the blowing

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through of carbon dioxide. Experiments have shown that continuous evacuation by means of a geared pump is possible with only a moderate vacuum. It must still be determined, however, whether the lactam can be sufficiently removed under a moderate vacuum by blowing carbon dioxide through. Experiments along this line are now being worked on in the laboratory.

Experiments on the effect of various stabilizers on the process and on the end product of evacuation were also worked on in the laboratory. The interesting observation was made that when the molten caprolactam polymerization product stabilized with 1/400 or 1/800 mole of acid was evacuated, of all the numerous acids used, only phosphoric, nitric, and sulfuric acid produced an abnormal stabilization curve. Details will be reported later.

Perfection of Textile Treatment of Perlon

For work on this subject a machine was developed in which perlon thread can simultaneously be stretched, boiled, dried, and perhaps even treated and wound. The machine is being built by the firm of Weinrich and Keller in Chemnitz-Einsiedel and is to be ready in a few weeks, providing we are able to overcome certain difficulties in the matter of procuring materials, gears, etc. It is planned first to produce perlon bristles and Leska wire in a continuous, single-stage process from spindle to finished product, and then to conduct experiments with both fine and coarse perlon thread (silk).

Development of a Process for Continuous Polymerization Perlon Under Pressure

For the experiments conducted in the DK [pressure?] tube during the third quarter, the arrangement used in the fifth series of experiments was retained except that the boiling stones used in the second stage of the double tube to achieve more intensive dehydration were removed, because these stones, which consisted of clay fragments, gradually disintegrated and caused great difficulties by clogging up cocks, pumps, and jets. For purposes of comparison, the apparatus was used with a pressure stage, that is, as a DK apparatus, and also without a pressure stage, as a VK [vacuum?] apparatus. With the use of 1/400 mole of acetic acid as stabilizer, viscosities up to η_{rel} = 2.5 were obtained. Since the Bosch pumps necessary for the pressure stage are not made in the German Democratic Republic, a simplified Bosch pump was developed with the assistance of the machine shop. The experiments conducted with this piston pump, which was built in the film factory, although they are not yet completed, show that the pump is serviceable for our purposes.

Except for the DK and VK experiments made with this DK apparatus, a trial arrangement with a combined VK/DK system was used, such as was included in our first VK patent application. To be sure, the pressure which the steam blowers exert on the lactam amounts to only about 0.2 atmosphere (gauge), corresponding to the column of molten lactam, about 2 meters. With very long polymerization periods, about 2 days, it has been possible to produce satisfactory fine filaments (silk) with this apparatus. With shorter processing periods, it appears that the use of AH salt or amino caproic acid, as projected in the patent application, is unavoidable. Experiments along this line are still being conducted. The spun silk produced in this apparatus with the use of a 45-percent aqueous solution of PT 1227 as treating solution was for the most part processed into fiber because our twist-stress machine was not set up ready for operation until the experiments were almost completed. The spools worked up as silk were tested for stretch distortion by the Technical Group on Artifical Silks (Kunstseiden-Technikum) and processed into knit pieces. Depending on the stretch ratio, the constants were as follows:

Nm	Rkm	Elongation (%)
140	55.6	13.8
113.8	47.6	28.6
111.2	47.1	21.2
113.8	42.6	38.1
108.8	40.1	34.6

Within the scope of this subject, the experiments on the effect of water on the continuous polymerization of caprolactam without pressure, and, in conjunction with this, the effect of the polymerization period and of the substances added to the liquid mass upon the formation of a product capable of being spun, were continued. These experiments confirm our former opinion concerning the great effect of the water from the AH salt or from the amino acid. As is well known, the polymerization of caprolactam can be speeded up extraordinarily by the addition of one percent of AH salt (1/232 mole), provided the water of condensation from the AH salt is not constantly removed. However, if nitrogen or carbon dioxide is introduced for quick removal of all traces of water, the experiments showed that even after 24 hours the liquid was still highly fluid and to a great extent water-soluble.

Testing of New Raw Materials for Suitability for Processing Into Completely Synthetic Fibers

During the third quarter the Buna Plant in Schkopau did not deliver any samples of amino capronitrile, AH salt, or hexamethylene diamine, because in their opinion it is not possible to produce completely pure polyamide raw materials on a laboratory scale. However, during the fourth quarter substantial deliveries are to be made of polyamide raw materials commercially produced.

As already reported, the samples of amino capronitrile received during the second quarter were not sufficiently pure for use in the production of perlon. We ourselves fractionated the last sample in four fractions, but did not succeed in obtaining amino capronitrile of sufficient purity for condensation.

As preparatory work for the future work to be done with large quantities of AH salt, the condensation conditions were again tested out in very small-scale experiments. Experiments were set up on the condensation periods required for AH salt, the condensation temperatures, the pressure, the sponginess of the liquid mass, etc. They proved that polyamide formation was considerably faster with AH salt with the use of high temperatures and the addition of water than with caprolactam. When AH salt was heated for 5 hours at 270-280 degrees with water, polyamide products of η rel = 2.3 were obtained. If pressures of only 4 - 5 atmospheres (gauge) were used, the viscosities in general were only η rel = 2.1. With 15 - 20 atmospheres (5 hours at 270-280 degrees the above-mentioned stage of η rel = 2.3 was reached.

Experiments were also initiated on producing the polyester terylene from the methyl ester of terephthalic acid and ethylene glycol. Since only small quantities of terephthalic acid were available, which were esterified by us in the usual way with methanol and hydrochloric acid, these experiments too were carried out only on a very small scale.

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